

LOW TEMPERATURE ANNEALING

OF IRRADIATED

COMMERCIALLY PURE TITANIUM

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by

LOCKHEED NUCLEAR PRODUCTS

C. A. Schwanbeck, Project Manager

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRA

Contract NAS 3-10298

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TOPICAL REPORT

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May 1968

Contract NAS 3-10298

Technical Management
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FOREWORD

This report is submitted to the National Aeronautics and Space Administration, Lewis Research Center, by the Lockheed-Georgia Company in accordance with the requirements of Article XXI, NASA Contract NAS 3-10298.

TABLE OF CONTENTS

Section		Page
Forward		iii
Table of Conten	nts	٧
List of Tables ar	nd Figures	vii
List of Symbols		ix
1	SUMMARY	1
2 .	INTRODUCTION	3
3	TEST MATERIAL	5
4	TEST SPECIMENS	5
5	TEST EQUIPMENT AND PROCEDURES	7
5.1	IRRADIATION TEST LOOP	7
5.2	TENSILE TESTING PROCEDURES	8
6	TEST RESULTS	9
6.1	TEST RESULTS FOR SPECIMENS TESTED AT 17°K AFTER POST-IRRADIATION ANNEALING	9
6.2	TEST RESULTS FOR SPECIMENS TESTED AT THE ANNEALING TEMPERATURE AFTER POST-IRRADIATION ANNEALING	9 DN
7	ANALYSIS OF RESULTS	11
8	CONCLUDING REMARKS	15
9	REFERENCES	17
10	TABLES -	19
11	FIGURES	23
APPENDIX A	SIGNIFICANCE OF DIFFERENCES BETWEEN THE SAMPLE MEANS	Α-
APPENDIX B	DISTRIBUTION	B-1

LIST OF TABLES AND FIGURES

Tables:		Page
1	Tensile Test Results, Titanium 55A (Annealed) Tested at 17°K After Stabilization at 17°K With Interim Warming To Indicated Temperature; Irradiation, As Indicated, At 17°K	21
II	Tensile Test Results, Titanium 55A (Annealed) Tested at Indicated Temperature After Stabilization at 17°K; Irradiation, As Indicated, At 17°K	22
A-1	Group Designations for Statistical Analysis	A- 3
A-II	Summary of Statistical Analyses of Significance of the Difference Between Group Mean Values of Tensile Ultimate Strength Data on Titanium 55A Presented in Tables I and II	A-4
A-III	Summary of Statistical Analyses of Significance of the Difference Between Group Mean Values of Tensile Yield Strength Data on Titanium 55A Presented in Tables I and II	A-5
A-IV	Summary of Statistical Analyses of Significance of the Difference Between Group Mean Values of Elongation Data on Titanium 55A Presented in Tables I and II	A-6
Figures:		
1	Tensile Specimen	25
2	Tensile Test Loop	26
3	Load Control System (Schematic)	27
4	Effects of Annealing Following Irradiation to $6 \times 10^{17} \text{ n/cm}^2$ At 17°K, Tested At 17°K, Titanium 55A (Annealed)	28
5	Effects of Testing Temperature Titanium 55A (Annealed), Tested Unirradiated and Following Irradiation to 6×10^{17} n/cm ² at 17°K	29
6	Effect of Annealing on Irradiation Induced Changes in Tensile Properties of Titanium 55A Irradiated to 6 x 10 ¹⁷ n/cm ² at 17 °K	30

LIST OF SYMBOLS

n/cm² fast fluence, in neutrons per square centimeter, with

energies above 0.5 MeV or 80 fJ

MeV million electron volts

fJ femtojoules

°K degrees Kelvin

Ksi thousands of pounds per square inch

kN/cm² kilo newtons per square centimeter

MN/cm² mega newtons per square centimeter

 F_{tu} Ultimate tensile strength

F_{ty} Tensile yield strength

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1 SUMMARY

This report describes the results obtained in an experimental study of the effects of post-irradiation annealing at cryogenic temperatures on the tensile properties of commercially pure titanium (Titanium 55A-Annealed). Test specimens were exposed to fluences of $6 \times 10^{17} \text{ n/cm}^2$, with neutrons energies greater than 0.5 MeV (80 femtojoules), at 17°K. The irradiated specimens were tested after annealing periods of one hour (3.6 x 10^3 sec) at 78°K and 178°K. Parallel sample lots were tested at the annealing temperature and after recooling to 17°K. Additional specimens were tested at 17°K following irradiation at 17°K with no post-irradiation annealing period.

The sample lots tested at 17°K following post-irradiation annealing showed that a pronounced reduction of the ultimate tensile strength occurred at temperatures below 78°K, while a less pronounced effect on the tensile yield strength occurred over the entire annealing temperature range.

The sample lots tested at the post-irradiation annealing temperature showed a gradual reduction in the irradiation effect on the ultimate tensile strength over the range of test temperatures. This reduction was substantial while there was relatively little reduction in the yield strength.

The residual effects of irradiation on the yield strength after annealing at 78°K and 178°K are, within the limits of experimental accuracy, independent of testing temperature. This contrasts with the results from aluminum similarly investigated in an earlier program.

2 INTRODUCTION

The concept of using hydrogen, stored in the liquid state, as the propellant for nuclear rockets to be used on trans-lunar missions is attractive because of the high specific impulses obtainable. Engineers responsible for the design of this sophisticated hardware require a knowledge of the combined effects of neutron irradiation and cryogenic temperatures on structural materials. Since lattice defects introduced by neutron irradiation are mobile even at liquid hydrogen temperature (20.5°K), irradiation and testing must be conducted at a temperature at least this low to observe effects which might occur in structural components of nuclear rockets.

Earlier test programs, conducted at the Plum Brook Reactor Facility of the NASA Lewis Research Center under Contracts NASw-114 and NAS3-7985, studied the following effects:

- The effect of fast neutron fluences* of 10¹⁷ n/cm² at 17°K on the tensile properties of thirty-three (33) metals and alloys including titanium alloys (ref. 1)
- The effect of fluences up to 10^{18} n/cm² at 17°K on the tensile properties of commercially pure titanium and several titanium alloys (ref. 2)
- The effect of fast neutron fluences of 10¹⁷ n/cm² at 17°K on the low-cycle fatigue properties of commercially pure titanium and two alloyed titaniums (ref. 2)
 - The effects of irradiation temperature and post-irradiation annealing on high purity aluminum (ref. 2).

The results obtained in the irradiation effects studies on Titanium 55A (annealed) conducted in the earlier programs (refs. 1 and 2) showed that a small but measurable increase in strength parameters exhibited a direct dependence on irradiation level for specimens irradiated and tested at $17\,^{\circ}\text{K}$. This was accompanied by a slight reduction in the ductility parameters. An increase in the F_{ty}/F_{tu} ratio was an observable radiation effect.

As a result of these programs, a further series of tests was authorized by Contract NAS3-10298. One series of tests from this program, a study of the effects of post-irradiation annealing on unalloyed titanium (Titanium 55A), has been completed. These tests included tensile tests on Titanium 55A, sample lots of three specimens, after irradiation to a fluence of 6 x 10^{17} n/cm² at 17° K followed by annealing periods of one hour (3.6 x 10^{3} sec) at 78°K and 178°K respectively. Parallel sample sets were tested at the annealing temperature and after recooling to 17° K. Control tests of unirradiated specimens were made after a similar thermal cycling exposure. The test results obtained are reported and discussed in the following sections of this report.

^{*} Neutron energies greater than 0.5 MeV or 80 femtoioules.

TEST MATERIAL

3

All test specimens were fabricated from a common lot of material; the same lot was used in the earlier test programs reported in references 1 and 2. According to the vendor, the Kroll process was used to produce elemental titanium from rutile (Ti O₂) through chlorination of a mixture of the ore and tar followed by reaction with metallic magnesium. The resultant sponge was vacuum melted in water cooled copper crucibles using consumable electrode techniques. Ingot reduction was accomplished through rolling with break down passes above the beta transus; finishing operations were in the alpha range. The finished material was annealed at 1300°F (980°K) for two hours (7.2 x 10³ sec) followed by cooling in still air. The test material had the following chemical composition:

<u>Fe</u>	<u>c</u>	-	<u>!</u>	<u> </u>	<u>+</u>	<u> </u>		2
wt%	wt%	At%	wt%	At%	wt%	At%	wt%	At%
0.19	0.032	0.13	0.023	0.08	0.006	0.29	0.218	0.65

with the remainder titanium.

4 TEST SPECIMENS

Due to space limitations in the irradiation access port (HB-2) and refrigeration capacity limitations, the specimen used was a miniaturization of the standard round specimen of ASTM E 8-66 (ref. 3), with a nominal gage diameter of 0.125 inch (0.318 cm) and a nominal gage length of 0.5 inch (1.27 cm). The tensile specimen used is shown in Figure 1. The ratios of the significant parameters are the same as for the standard ASTM specimen. Unirradiated control specimens were run for each thermal environment of the irradiation testing program to ensure a common basis for the evaluation of irradiation effects.

5 TEST EQUIPMENT AND PROCEDURES

The studies of annealing effects performed in this experiment fall into two general cases:

- 1. Specimen irradiated to 6×10^{17} n/cm² at 17° K, annealed for 1 hour (3.6 × 10^{3} sec) at higher cryogenic temperatures (78°K and 178°K), re-cooled to 17°K for 1 hour (3.6 × 10^{3} sec) and tested at 17°K.
- 2. Specimen irradiated to 6×10^{17} n/cm² at 17° K, annealed for 1 hour (3.6 x 10^{3} sec) at higher cryogenic temperatures and tested at the annealing temperatures.

Unirradiated control specimens were tested after low temperature thermal cycling identical to that received by the irradiated specimens except that the initial exposure at 17° K was for one hour (3.6 x 10^3 sec) rather than the 75 to 110 hour (2.7 x 10^5 to 4.0 x 10^5 sec) at 17° K required to achieve a fluence of 6 x 10^{17} n/cm² in HB-2.

All testing was conducted, as nearly as feasible, in accordance with American Society of Testing Materials Specifications ASTM E 184-62 and E 8-66 (ref. 3).

5.1 IRRADIATION TEST LOOP

All testing was performed at the NASA Plum Brook Reactor Facility using the horizontal beam port on the north face of the reactor core, designated as HB-2, as the irradiation facility. The testing machines are contained in cryogenic test loops capable of insertion into the 6" (15.24 cm) diameter beam port. Transfer tables provide the capability of insertion and withdrawal of the loops from HB-2 and provide rotation to permit positioning the loops in a radially aligned hot cave for specimen change. Specimen temperature control is maintained with an 1150 watt refrigerator using helium as the cryogenic fluid. Detailed descriptions of the test hardware may be found in references 1 and 2. A drawing of the test loop is shown in Figure 2. The load control system, shown schematically in Figure 3, permits axial loading of the specimen in tension or compression with applied forces up to 5000 lbs (22,240 newtons).

The temperature of the test specimen was controlled by platinum resistance sensors located in the inlet and outlet refrigerant lines. The validity of this method of control had been verified in an earlier program by comparison of the readings of these sensors with especially calibrated thermocouples affixed to a test specimen held in the specimen location of the test loop at all temperatures of interest, both in-pile and out-of-pile (refs. 1 and 2).

Control of the irradiation fluence was based on calculations made from the reactor power level and the control rod bank height. This method was established by threshold foil measurements made during the earlier programs and is described in detail in references 1 and 2.

5.2 TENSILE TESTING PROCEDURES

Tensile test methods conformed as nearly as possible to ASTM E 8-66 (ref. 3). The load rate was monitored, during elastic behavior of the specimen, by controlling the incremental strain at less than 0.0015 in/in/min (2.5 x 10⁻⁵/sec). The load was monitored with a proving ring type dynamometer calibrated to within two percent of a National Bureau of Standards certified reed type proving ring. The extensometer used was classified as ASTM E 83-64T, class B-2 under actual operational conditions. Extensometers of this classification, while adequate for the determination of the yield strength of metallic materials, are not normally used for measuring the modulus of elasticity or deviation from Hooke's Law. Therefore, the modulus values included in this report should not be considered as absolute values.

The principal departure from the ASTM testing procedures is in specimen geometry, discussed in Section 5.1.

Ductility parameters, elongation in 4D, and reduction of area were obtained by post-testing measurements of failed specimens in accordance with paragraphs 26 and 27 of ASTM E 8-66 (ref. 3).

The F_{ty} was obtained by the 0.2% offset method.

The fracture stress was obtained by dividing the load at fracture by the cross-sectional area of the failed specimen at the point of fracture. The data required for correcting this stress for the tri-axial state of stress during plastic instability are not available; therefore, this parameter as reported is of questionable reliability (ref. 4, p 246).

The accuracy and calibration of the test system are discussed in references 1 and 2.

6 TEST RESULTS

The test results for the test program are given in Tables I and II and shown graphically in Figures 4 and 5.

In both cases, the data from the control specimens are given and plotted with the results from the irradiated specimens.

6.1 TEST RESULTS FOR SPECIMENS TESTED AT 17°K AFTER POST-IRRADIATION ANNEALING

The test data obtained from tensile specimens tested at 17°K following annealing at 78°K and 178°K of effects induced by irradiation to $6 \times 10^{17} \text{ n/cm}^2$ at 17°K are shown in Table I and Figure 4.

Examination of Figure 4 shows that the principal annealing effect in the F_{tu} occurs between 17°K and 78°K while the effect on the F_{ty} appears fairly linear with temperature over the range from 17°K to 175°K. This is also indicated by the convergence at both end points of the curves showing the F_{ty}/F_{tu} ratio at the several temperatures. The effect of annealing on the ductility parameters appears to be negligible in this temperature range.

There was no apparent irradiation effect on the fracture stress.

6.2 TEST RESULTS FOR SPECIMENS TESTED AT THE ANNEALING TEMPERATURE AFTER POST-IRRADIATION ANNEALING

The test data obtained from tensile specimens tested at the annealing temperature following post-irradiation annealing at 78°K and 178°K after irradiation to 6×10^{17} n/cm² at 17°K are shown in Table II and Figure 5.

Examination of Figure 5 shows that the annealing of the effect of irradiation on the F_{tu} is more nearly linear with respect to temperature over the range from 17 °K to 178 °K than was noted for the specimen tested at 17 °K following similar post-irradiation anneals. The gradual convergence of the curves plotting the F_{tu} values for unirradiated and irradiated specimens with increasing annealing and test temperatures shows an essentially linear mitigation of the radiation effects on the F_{tu} as a function of temperature. The curve of the effect of annealing and test temperature on the F_{ty} shows no such convergence; the unirradiated and irradiated specimens produce parallel

curves. This causes a divergence of the F_{ty}/F_{tu} ratio curves with increasing temperature. The cryogenic effects on the ductility parameters appear to be of greater significance than the minor irradiation induced embrittlement observed in the curves. As with the specimen post-anneal tested at $17\,^{\circ}$ K, the annealing of ductility parameters seems slight. The apparent maxima near $80\,^{\circ}$ K, observable on both ductility parameters, is a cryogenic effect unrelated to irradiation. Similar peaks have been reported by other investigators working on the cryogenic behavior of titanium and titanium alloys (ref. 5 and 6).

There was no apparent irradiation effect on the fracture stress.

ANALYSIS OF TEST RESULTS

7

The statistical significance of differences between various sample means of several tensile properties reported in Tables I and II were calculated using the method described in Appendix A. Results of these calculations are summarized in tabular form in this appendix. Other properties reported in Tables I and II were not statistically analyzed; the Fty/Ftu ratio was calculated from values that were analyzed, the modulus and the fracture stress values were derived from measurements of insufficient exactness to warrant statistical analysis, and the reduction of area is of little engineering interest and is a rather imprecise value used primarily as a measure of material quality. Although in this program only three specimens were tested for each set of conditions, these analyses show that in only a few cases is there a significant probability that the testing of additional specimens might have changed the statistical significance of the results.

Statistical analysis of the data obtained from unirradiated specimens yields the anticipated results; there is no significant difference in the mechanical properties of any sample lots tested at 17 °K regardless of prior cryogenic thermal cycling and statistically significant and large effects of test temperature are observable. The observed increase in the strength functions due to neutron irradiation are verified statistically. Substantial residual irradiation strengthening is observable after post-irradiation annealing when tested at either the annealing temperature or after recooling to 17 °K.

Comparison of the F_{tu} values of the irradiated specimens tested at 17°K following post-irradiation anneals at 78°K and 178°K show that the major portion of the annealing has occurred at temperatures below 78°K; the difference in the values for this parameter after annealing at 78°K and 178°K is not significant statistically at the 90% confidence level.

The changes in the F_{ty} values in the same sets of specimens show an essentially linear response to annealing temperature. Although the net change in the parameter after annealing at 78 °K is of marginal statistical validity at the 90% confidence level, the plot of arithmetic means (fig. 4) indicates that the annealing effect on the F_{ty} is probably uniform over the temperature range investigated. This is one instance where a larger sample lot might be expected to verify a trend obscured by data scatter in a small specimen sample.

Statistical comparisons of the elongation data presented in Table I show that annealing of either irradiated or unirradiated material at 78 or 178°K followed by tests at 17°K does not significantly change the amount of elongation from that of material tested at 17°K without intermediate anneals at 78 or 178°K. A small (about 3%) but statistically significant reduction in the amount of elongation occurred in the material irradiated and tested at 17°K. The slight reductions in elongation recorded for irradiated

samples annealed at 78 or 178°K prior to testing at 17°K are not statistically significant. Very small reductions in elongation in the irradiated samples tested at the annealing temperatures of 78 and 178°K were recorded. The reduction at 78° is statistically significant but that at 178° is not.

Variations in test temperatures produce apparent differences in the amount of reduction of area in Titanium 55A during tensile tests. No apparent effects of radiation on this property were obtained in this program.

Figure 6 is a graphical presentation of the effects of annealing and test temperatures on radiation induced changes in the strength parameters of titanium. The mean difference between groups of irradiated and unirradiated specimens tested under each set of thermal conditions is plotted with one standard deviation on either side of the mean indicated. This difference is the residual radiation effect on these properties representing an increase in strength. The magnitude of the standard deviation for the Ftu data point for the specimens tested at 17°K following post-irradiation annealing at 178°K might lead to misleading interpretation of these data in Figure 6. Since, due to theoretical considerations, this plot is not likely to be concave upwards, the curve must be biased intuitively to provide a meaningful plot. The data points are not, of course, absolute values; they are measured values and allowance must be made for experimental uncertainty in evaluation. Such data can not be handled in a purely statistical manner. Consideration of probable solid state and metallurgical mechanisms must be given in data interpretation.

In general, the irradiation induced increase in the ultimate tensile strength, while not as large as that in the yield strength, is annealed more rapidly than the increase in the yield strength. This is true regardless of testing temperature for annealing at 78 °K and true for the measurement at the annealing temperature of 178 °K. This results in a higher yield to ultimate ratio (0.917) for annealing and testing at 178 °K than for any other test condition.

The absolute decrease in the residual effect on annealing at 178°K and testing at 17°K is about the same in the ultimate tensile strength as in the yield strength.

The residual effect, in any case, regardless of parameter, annealing, or test temperature is no less than 30 percent of the irradiation induced increase at 17 °K without annealing.

A more surprising result is that the residual effects on the yield strength are, within the limits of experimental accuracy, independent of the test temperature. In terms of dislocation theory, this means that defects introduced by the neutron irradiation at 17 °K and remaining at temperatures up to 178 °K act as obstacles to plastic flow that cannot be overcome by thermal fluctuations up to 178 °K (ref. 6). Such obstacles are assumed to be large (greater than 20×10^{-10} meters [ref. 7]) and to have stress fields like those associated with large precipitates or dislocations on intersecting slip planes. They affect the temperature dependence only indirectly and they might be expected to form and remain in titanium under the described test conditions.

At the same time, with regard to the other residual effects, the combined effects of recombination of close vacancy-interstitial pairs and interstitial migration to trapping sites (ref. 8) at temperatures up to 178°K might be expected to reduce the irradiation effect on the yield strength. And it can be argued that this diffusion of defects could reduce the irradiation effect on the ultimate strength even more by eliminating some of the sources of work hardening. The fact that this is not indicated by the ductility data, which if anything indicate an increase in hardening sources, at least for tests at 17°K, might be attributed to the limited precision of these measurements.

There are certain differences between the irradiation annealing effects in titanium and the irradiation annealing effects reported for aluminum in reference 2. These differences are attributable to differences in the unirradiated materials and differences in the effects of irradiation before annealing as well as to differences in annealing phenomena. The most apparent differences between titanium and aluminum are in the residual irradiation effects. Annealing is much slower in titanium than in aluminum for a given annealing temperature. Also temperature dependence of the residual effects is less pronounced than in aluminum. Both of these effects might be related to the much higher melting point of titanium. A lower defect diffusion rate would be expected in titanium and the number and distribution of defects before annealing would be expected to differ from aluminum at least because of the higher melting point and possibly because of other fundamental differences such as crystal structures and grain sizes.

Serrated yielding, reported by other investigations at 4°K (ref. 6) was not observed in the load-elongation curves obtained from cryogenic testing of titanium in this program. The lowest test temperature in this program, 17°K, is 0.009 of the absolute melting temperature, that used by Kula in the work reported in reference 6 was 0.002 of the melting temperature. The pronounced increase in resistance to plastic deformation observed in all body centered cubic and some hexagonal close packed lattices at extremely low temperatures (ref. 10) may occur between these two temperatures in titanium.

8 CONCLUDING REMARKS

Tensile testing of commercially pure titanium has been performed under various test conditions after irradiation to 6×10^{17} n/cm² (with energies greater than 80 fJ) at $17\,^{\circ}$ K. Tensile test characteristics were obtained at the irradiation temperature after one hour $(3.6 \times 10^3 \text{ sec})$ at $78\,^{\circ}$ K and after one hour at $178\,^{\circ}$ K. Tensile measurements were also made at the two annealing temperatures following the same irradiation and annealing. These results along with those from similarly irradiated specimens without annealing have been compared to results from unirradiated specimens at the same test temperatures.

Irradiation to 6×10^{17} n/cm² at 17 °K, without annealing, increases the ultimate tensile strength of this material by about 10%, the tensile yield strength by about 20%, over the values obtained at 17 °K without irradiation. A slight irradiation induced reduction in the values of the ductility parameters is observable.

Testing at 17 °K following post-irradiation annealing at 78 °K showed a distinct diminution in the irradiation induced increase in the ultimate tensile strength. The annealing effect on the tensile yield strength of these specimens was less pronounced. Testing at 17 °K following post-irradiation annealing at 178 °K showed about equal reductions of both of these strength parameters. These data verify the importance of maintaining the temperature of interest throughout irradiation and testing periods, and all intervening time between these periods, in cryogenic-irradiation effect studies.

Testing at the annealing temperatures, following post-irradiation anneals at 78°K and 178°K, showed a substantial temperature dependent reduction on the ultimate tensile strength with relatively little annealing effect observed on the yield strength.

Residual irradiation induced increases in both strength parameters remained regardless of the annealing and test temperatures. The smallest remaining residual effect was in the ultimate tensile strength after annealing at 178 °K and testing at the annealing temperature. This residual increase was only about 30 percent of the increase measured at 17 °K without annealing. Other residual increases after annealing at 178 °K and measuring at either 178 °K or 17 °K were equal to about 50 percent of the effects measured without annealing.

There were little, if any, annealing effects on the ductility parameters, regardless of the annealing and test temperatures.

There are certain differences between irradiation annealing effects in titanium and those in aluminum, the most obvious being much larger residual effects in the strength parameters in titanium than in aluminum. Also, these residual effects are less test-temperature dependent in titanium than in aluminum.

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TABLES

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	kN/cm ²	92.6	90.3	87.2	90.05	8	72.7	93.6	4.06	92.30	92.5	90.5	92.5	91.82	106	901	109	107.1	101	105	2	104.6	001	102	101	101.2
u.	Ksi	134	131	126	130.6		3	136	131	133.9	134	131	35	133.2	154	15.	158	155.3	152	152	151	151.7	145	149	146	146.7
	kN/cm ²	129	125	127	127.3		<u></u>	127	127	127.7	129	122	127	125.6	145	9	=	142.0	131	134	135	133.5	132	134	134	133.4
L.	Ks:	188	182	185	184.7	•	88	184	184	185.2	186	176	184	182.1	1112	203	20.	206.0	180	195	196	193.6	16	195	19.5	193.4
1	n/cm ²	None None	None	None	None	:	None	None	None	No.	None	None	None	None	7101 4 9	6 × 10 ¹⁷	6 × 1017	6×10^{17}	6×10^{17}	6× 1017	6 × 10 ¹⁷	6 × 1017	71 ⁰¹ × 9	201 29	51 01 × 9	6× 1017
<u> </u>		11	17	17	17	i	8	78	78	78	178	178	178	178	17	: 1	. 21	11	78	78	78	78	178	178	178	178
	Specimen	1 Ag 2	1 Ag 3	1 Ag 211	Mean *		1 Ag 16	1 Ag 19	1 Ag 34	Mean *	1 Aa 24	1 Ag 25	2 1 Ag 53		1 42 153 (a)	1 Aa 200 (a)	1 Ag 203 (a)	Mean *] Ag 4	1 Ag 14	1 Ag 47	Mean *	1 ♣2 28	1 40 41	1 Ag 50	Mean *

TENSILE TEST RESULTS, TITANIUM 55A (ANNEALED) TESTED AT 17°K AFTER STABILIZATION AT 17°K WITH INTERIM WARMING TO INDICATED TEMPERATURE, IRRADIATION, AS INDICATED, AT 17°K

TABLE I

Means obtained before rounding Ratios obtained before rounding Means are of ratios

Previously reported in ref. 2. For Comparison Purposes Only Not determinable **©*** ₁

TABLE II

TENSILE TEST RESULTS, TITANIUM 55A (ANNEALED) TESTED AT INDICATED TEMPERATURE AFTER STABILIZATION AT 17°K; IRRADIATION, AS INDICATED, AT 17°K

Modulus (♣)	MN/cm ²	12-13	2242	15 12 12 12	01-8	10-14	12 7 2 1	5725
,	10 ³ Ksi	18-19 19	18 17 20 18	15 21 17 18	12-14	15-20 18	19 20 18 19	17 20 18 18
Fracture Stress	KN/cm ²	226-243 235.8	225 203 207 211.7	125 164 128 139.1	ı ı	235-262 250.8	210 202 208 206.8	138 132 132 133.8
Fractor	Ksi	328-352 342.0	327 294 300 307.0	181 238 186 201.7	1 1	341-380 363.7	305 293 302 300.0	200 191 191 194.0
Reduction of Area	%	44-48 46.3	67 68 65 67.0	61 62 62.3	59-65 62.3	38-46 43.0	63 63 64.0	2 2 2 2 2 2 7.2 2 7.2 2 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1
Elongation (4D)	%	30-32 31.0	47 51 52 50.0	35 26 30 30.3			39 41 41.0	25 29 30 28.0
н Н	· • • • •	0.68-0.72	0.76 0.78 0.76 0.76	0.84 0.84 0.83	0.73-0.91 0.798	0.73-0.78 0.753	0.83 0.85 0.80 0.827	0.90 0.92 0.93 0.917
	kN/cm ²	87.2-92.6 90.05	72.9 73.2 70.8 72.31	55.1 54.4 53.0 54.17	32.8-43.6 36.89	106-109	82.9 86.6 79.8 83.13	62.5 63.9 63.8 63.43
ui	Ksi	126-134 130.6	106 106 103 104.9	79.9 78.9 76.9 78.57	47.5-63.3 53.50		120 126 116 120.6	%.7 %2.7 %2.6 %2.00
	kN/cm ²	125-129 127.3	95.3 94.2 92.7 94.07	95.2 65.6 94.3 65.0 94.2 65.0 94.57 65.21	44.9-47.9 46.20	140-145 142.0	99.8 101.4 99.8 100.3	69.5 69.5 68.9 69.32
ú	Ksi Fi	182-188 184.7	138 137 134 136.4	95.2 94.3 94.57	65.1-69.4 67.00	203. 211 206.0	145 147 145 145.5	101 101 100 100.5
(; t ; t ; t ; t ; t ; t ; t ; t ; t ;	n/cm ²	None None	Z Z Z Z Z Z Z Z Z Z Z Z Z Z Z Z Z Z Z	None Zone	None None	6 × 10 ¹⁷ 6 × 10 ¹⁷	6 × 1017 6 × 1017 6 × 1017 6 × 1017	6 × 1017 6 × 1017 6 × 1017 6 × 1017
<u>.</u>	E ¥	71	78 78 78 78	178 178 178 178	88	71	78 78 78 78	178 178 178 178
	Specimen	Range of 3(b) Mean of 3(b)	1 Ag 11 1 Ag 195 1 Ag 201 Mean *	1 Aa 17 1 Aa 51 1 Aa 59 Mean *	Range of 5(c) Mean of 5(c)	Range of 3(b) Mean of 3(b)	1 Aa 1 1 Aa 42 1 Aa 212 Meen *	1 Aa 23 1 Aa 35 1 Aa 45 Meen *

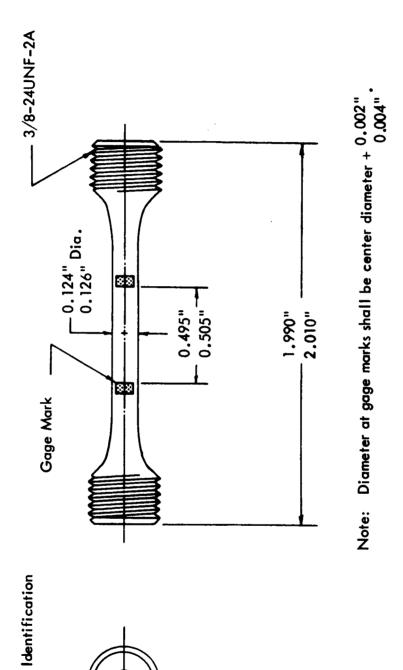
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Means obtained before rounding Ratios obtained before rounding Means are of ratios

. :

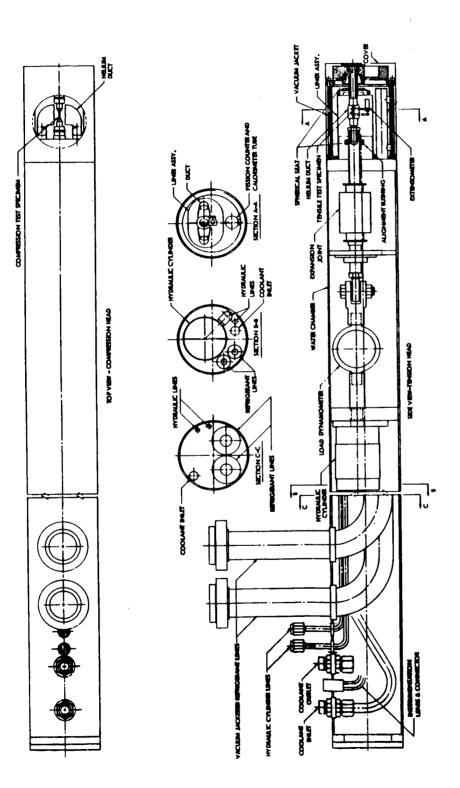
From Table 1 Previously reported in ref. 1 For comparison purposes only Not determinable

11 FIGURES



TENSILE SPECIMEN

FIGURE 1 TI



TENSILE TEST LOOP

FIGURE 2

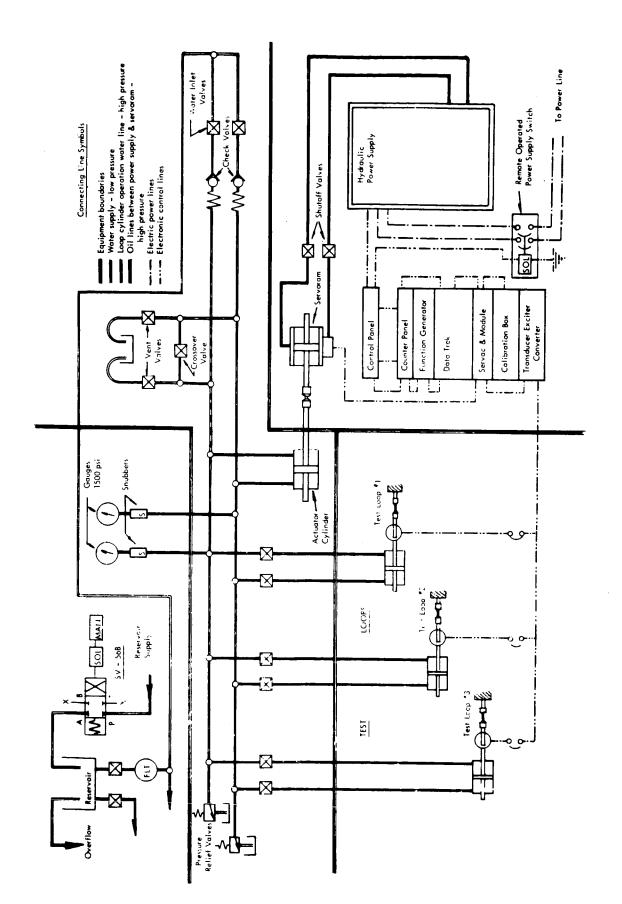
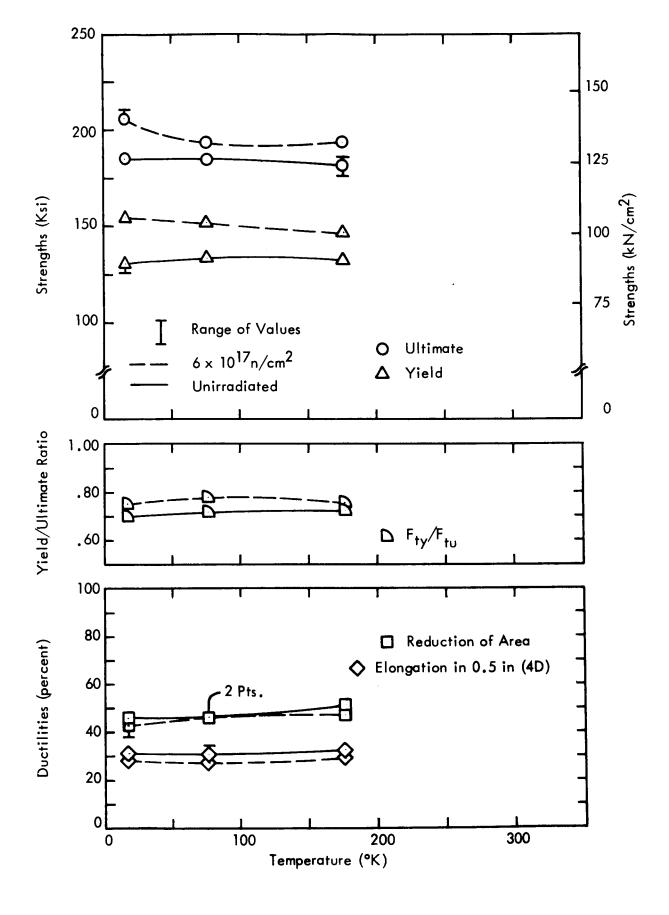


FIGURE 3 LOAD CONTROL SYSTEM (SCHEMATIC)



EFFECTS OF ANNEALING FOLLOWING IRRADIATION TO 6 x 10¹⁷ n/cm² AT 17°K, TESTED AT 17°K, TITANIUM 55A (ANNEALED)

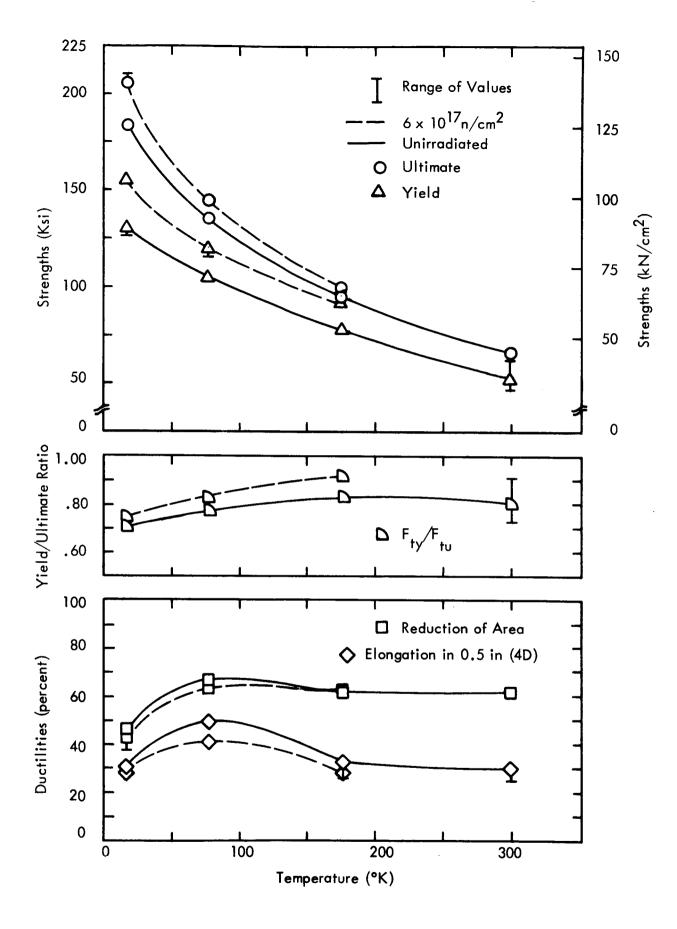


FIGURE 5 EFFECTS OF TESTING TEMPERATURE TITANIUM 55A (ANNEALED), TESTED UNIRRADIATED AND FOLLOWING IRRADIATION TO 6 × 10 17 n/cm² AT 17°K

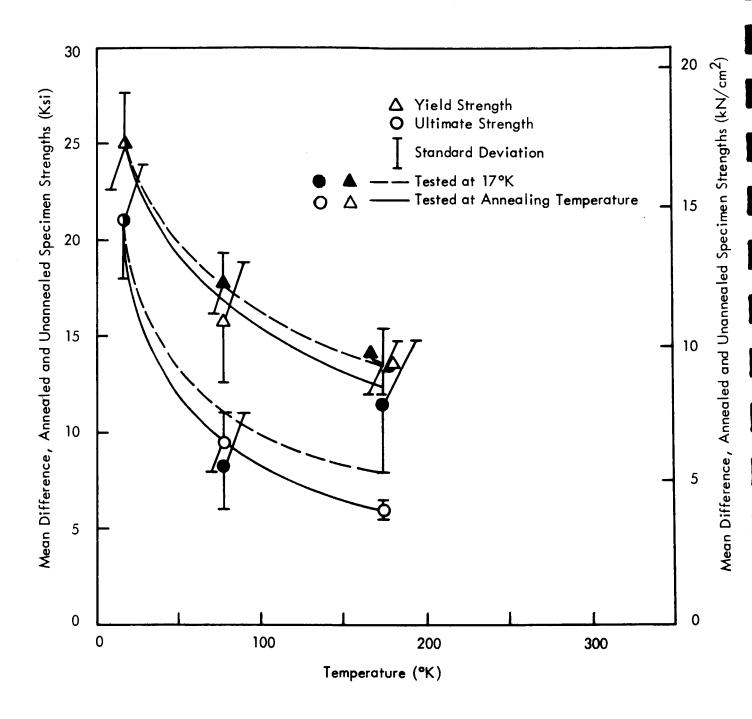


FIGURE 6 EFFECT OF ANNEALING ON IRRADIATION INDUCED CHANGES IN TENSILE PROPERTIES OF TITANIUM 55A IRRADIATED TO 6 x 10¹⁷ n/cm² AT 17°K

APPENDIX A

SIGNIFICANCE OF THE DIFFERENCE BETWEEN TWO SAMPLE MEANS

To determine if the difference between the means of two groups of samples is statistically significant the null hypothesis, that the two sample means \vec{X}_1 and \vec{X}_2 are from the same population with respect to the population mean \vec{X}_p , is used. This hypothesis is tested by determining the probability of t, where t is the ratio of $\vec{X}_1 - \vec{X}_2$ to an estimate of the standard error of the difference between the two sample means.

The standard error of the difference between two sample means, $\sigma_{\bar{\chi}_1 - \bar{\chi}_2}$ is given by:

$$\sigma_{\bar{X}_1 - \bar{X}_2} = \sigma \sqrt{\frac{1}{N_1} + \frac{1}{N_2}}$$
 (1)

where σ is the standard deviation of the population and N_1 and N_2 are the number of items in sample one and sample two, respectively.

Since the value of σ is unknown, its value must be estimated from the information given by the two samples. This estimate is $\tilde{\sigma}_{1+2}$ obtained from:

$$\hat{\sigma}_{1+2} = \sqrt{\frac{\sum_{1}^{2} + \sum_{2}^{2}}{N_{1} - 1 + N_{2} - 1}}$$
(2)

 $\sum_{1}^{2} x_{1}^{2}$ and $\sum_{2}^{2} x_{2}^{2}$ can be obtained by:

$$\sum x^2 = \sum X^2 - \frac{\left(\sum X\right)^2}{N} \tag{3}$$

where X is the parameter value of each of the N items in the sample.

Having determined the value of ∂_{1+2} with equations (3) and (2), an estimate of the standard error of the difference between the two means is obtained from:

$$\hat{\sigma}_{\bar{X}_1 - \bar{X}_2} = \hat{\sigma}_{1+2} \sqrt{\frac{1}{N_1} + \frac{1}{N_2}}$$
 (4)

Equation (4) is derived from equation (1).

Finally the desired significance ratio t is obtained from:

$$t = \frac{\bar{X}_1 - \bar{X}_2}{\hat{\sigma}_{\bar{X}_1 - \bar{X}_2}}$$

This value of t and a t-distribution table (available in most statistics textbooks) are used to obtain the probability (P) of obtaining a value equal to $\frac{1}{2}$ t or more. The degrees of freedom n in this case is:

$$n = N_1 - 1 + N_2 - 1$$

Since one degree of freedom was lost when $\sum x_1^2$ was computed about \bar{X}_1 and another degree of freedom was lost when $\sum x_2^2$ was computed about \bar{X}_2 .

Tables A-II, A-III and A-IV present a summary of statistical evaluation of the data for the strength function and the elongation values presented in table I and II. The group designation are defined as shown in Table A-I.

TABLE A-1

GROUP DESIGNATIONS FOR STATISTICAL ANALYSIS

Group	Irradiation Exposure n/cm ² x 10 ¹¹⁷	Tempero Irradiation	ature, Degrees K Interim	Test
Α	0	*	None	17
В	0	*	78	17
С	0	*	178	1 <i>7</i>
D	6	17	None	1 <i>7</i>
Е	6	17	78	1 <i>7</i>
F	6	17	178	1 <i>7</i>
G	0	*	78	78
Н	0	*	178	178
I	6	17	78	78
J	6	17	178	178

^{*}All unirradiated specimens stabilized at 17°K prior to warm-up to interim temperature

TABLE A-II

SUMMARY OF STATISTICAL ANALYSES OF SIGNIFICANCE OF THE DIFFERENCE BETWEEN GROUP MEAN VALUES OF TENSILE ULTIMATE STRENGTH DATA ON TITANIUM 55A PRESENTED IN TABLES I AND II

Statistical Parameters	6 •	(•	•	G.	Groups Compared	ц « ц	ر م	а 4	ر «ر π
	A & B 2.69	4.30	4.09	3.84	3.30	2.55	3.74	2.87	3.90
	2.20	3.51	3.34	3.14	2.68	2.08	3.06	2.35	3,19
	0.5	2.6	3.1	12.4	12.6	0.2	21.3	8.4	11,3
	.227	.741	.928	3.95	4.70	960.0	96.9	3.57	3,54
	.80	0.50	.40	.02	.01	1.0	(D)	.03	.03
	Š	Š	Š	≺es	Yes	Š	≺es	Yes	Kes
				Gro	Groups Compared	Pe			
	A & G	A & H	G & H	٦ & ٦	D & J	ر ۾ ا	A & D	_ ଷ -	_ % ∓
	2.60	2.18	1.58	3.20	3, 12	1.00	3.74	1.73	707.
	2.12	1.78	1.29	2.61	2,55	.817	3.06	1.41	.577
	48.3	70.1	41.8	60.5	105.5	45.0	21.3	9.1	5.9
	22.8	50.6	32.4	23.2	41.4	55.1	96.9	6.45	10.2
	(a)	(p)	(a)	(p)	(a)	(a)	(a)	(a)	(°)
	Yes	Yes	Yes	Yes	Yes	Yes	Yes	≺es	Yes

(a) 0 < P < .01

TABLE A-III

SUMMARY OF STATISTICAL ANALYSES OF SIGNIFICANCE OF THE DIFFERENCE BETWEEN GROUP MEAN VALUES OF TENSILE YIELD STRENGTH DATA ON TITANIUM 55A PRESENTED IN TABLES I AND II

Statistical				Gro	Groups Compared	eq			
rarameters	A & B	A & C	8 8 C	D & E	D & F	Е & Т	A & D	8 & E	О 8
\vec{Q}_{1+2}	3.43	3.12	2.24	1.73	2.24	1.58	3,32	1.94	1.94
$\vec{O}\vec{X}_1 - \vec{X}_2$	2.80	2.55	1.83	1.41	1.83	1.29	2.71	1.58	1.58
$ar{ar{\chi}}_1 - ar{ar{\chi}}_2$	3.3	2.9	7.0	3.6	8.6	5.0	24.7	17.8	13.5
+	1.18	1.14	.382	2.55	4.70	3.88	9.11	11.3	8,54
۵.	.30	.30	.70	.07	.01	.02	(a)	(a)	(D)
Significant at P = .05	Š	° Z	Š	°Z	Yes	Yes	Yes	> 8	Yes
Statistical				Grou	Groups Compared	- 0			
	A&G	A & H	G & H	٥ ۾ ا	D & J	_ & _	A&D	ი - გ	H & J
ر م ا	3.12	2.08	1.66	3.94	1.87	3.68	3.32	3.78	1.41
$\vec{\sigma}_{ar{\chi}_1^-}ar{\chi}_2^-$	2.55	2.52	1.35	3.21	1.53	3.00	2.71	3.08	1.16
$ar{oldsymbol{\chi}}_{1}$ – $ar{oldsymbol{\chi}}_{2}$	25.7	52.0	26.3	34.7	63.3	28.6	24.7	15.7	13.4
+	10.1	20.6	19.5	10.8	41.4	9.53	9.11	5.10	11.6
a	(a)	(a)	(a)	(o)	(o)	(o)	(a)	(a)	(a)
Significant at P = "05	Yes	≺es	Yes	Yes	Yes	Yes	Yes	≺es	Yes

(a) 0 < P < .01

TABLE A-IV

SUMMARY OF STATISTICAL ANALYSES OF SIGNIFICANCE OF THE DIFFERENCE BETWEEN GROUP MEAN VALUES OF ELONGATION DATA ON TITANIUM 55A PRESENTED IN TABLES I AND II

Statistical				Grou	Groups Compared	þ			
Parameters	A & B	A&C	B & C	D & E	D&F	E & F	A&D	В & Е	C R F
Q ₁₊₂	2.65	2.00	3.16	2.29	1.41	2.45	1.12	3,35	2.18
$\partial ar{\chi}_{l} - ar{\chi}_2$	2.16	1.63	2.58	1.87	1.16	2.00	.913	2.74	1.78
$ar{m{\mathcal{X}}}_1 - ar{m{\mathcal{X}}}_2$	0	2.0	2.0	1.6	0.4	2.0	2.7	4.3	4. S.
-	0	1.22	.775	.855	.346	1.00	2.96	1.57	2.42
a .	1.00	%	.50	.45	.75	.40	.04	.20	20.
Significant at P = .05	ž	°Z	ž	Š	°Z	°Z	Yes	ž	ĝ
Statistical				Grou	Groups Compared	70			
Parameters	A&G	A R H	G & H	D & I	D & J	ر ه ا	A&D	ଜୁଷ	H & J
<i>ر</i> م ا+،	2.00	3.28	3.71	1.66	2.06	2.34	1.12	2.34	3.71
$\hat{Q}_{X,-X_2}$	1.63	2.68	3.03	1.34	1.68	1.92	.913	1.92	3.03
$\vec{\mathbf{x}}_{1}$ $\vec{\mathbf{x}}_{2}$	19.0	7.0	19.7	12.7	0.3	13.0	2.7	9.0	2.3
· . +-	11.6	.262	6.51	9.38	.178	6.79	2.96	4.70	.760
a .	(a)	.80	(a)	(p)	.85	(a)	.04	.01	.50
Significant at P = .05	Yes	ŝ	Yes	Yes	° Ž	Yes	≺es	Yes	Š

APPENDIX B

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ABSTRACT

Commercially pure titanium (Titanium 55A - Annealed) was tested in tension after post-irradiation annealing at 78°K and 178°K following irradiation to $6 \times 10^{17} \text{ n/cm}^2$ (E > 0.5 MeV, 80 fJ) at 17°K. Tests were performed both at the annealing temperature and after re-cooling to 17°K. Post-irradiation annealing at 78°K or 178°K prior to testing at either the annealing temperature or at 17°K substantially reduced the radiation induced strength increases but did not appreciably affect ductility properties.